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ance Notes on Codes and Abbreviations" appearing at the begin-
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(54) Title: OPTIMIZING NANO-FILLER PERFORMANCE IN POLYMERS

(57) Abstract: A polymer composite comprising a polymer matrix having, dispersed therein, a nano clay in combination with a crosslinking promotor. In method form, the invention relates to a process for enhancing the mechanical properties of a polymer composite, comprising supplying a polymer matrix, combining the matrix with a nano clay and a crosslinking promotor and crosslinking.

WO 01/34685 A1

1 **OPTIMIZING NANO-FILLER PERFORMANCE**
2 **IN POLYMERS**

3 The present invention relates to nano clays for use in
4 thermoplastic/thermoset polymer materials, wherein the nano clay may be
5 combined with another chemical ingredient, such as a crosslinking agent, to
6 thereby provide a unique and overall synergistic effect on mechanical
7 property performance.

8 Polymer composites comprising a polymer matrix having one or more
9 additives such as a particulate or fiber material dispersed throughout the
10 continuous polymer matrix are well known. The additive is often added to
11 enhance one or more properties of the polymer. Useful additives include
12 inorganic layered materials such as talc, clays and mica of micron size.

13 A number of techniques have been described for dispersing the
14 inorganic layered material into a polymer matrix. It has been suggested to
15 disperse individual layers, e.g., platelets, of the layered inorganic material,
16 throughout the polymer. However, without some additional treatment, the
17 polymer will not infiltrate into the space between the layers of the additive
18 sufficiently and the layers of the layered inorganic material will not be
19 sufficiently uniformly dispersed in the polymer.

20 To provide a more uniform dispersion, as described in U.S. Pat. No.
21 4,889,895 sodium or potassium ions normally present in natural forms of
22 mica-type silicates and other multilayered particulate materials are exchanged
23 with organic cations (e.g., alkylammonium ions or suitably functionalized
24 organosilanes) thereby intercalating the individual layers of the multilayered
25 materials, generally by ionic exchange of sodium or potassium ions. This
26 intercalation can render the normally hydrophilic mica-type silicates
27 organophilic and expand its interlayer distance. Subsequently, the layered
28 material (conventionally referred to as "nanofillers") is mixed with a monomer
29 and/or oligomer of the polymer and the monomer or oligomer polymerized.

1 The intercalated silicate is described as having a layer thickness of 7 to 12
2 [Angstrom] and an interlayer distance of 30 [Angstrom] or above.

3 In WO 93/11190, an alternative method for forming a composite is
4 described in which an intercalated layered, particulate material having
5 reactive organosilane compounds is dispersed in a thermoplastic polymer or
6 vulcanizable rubber. Yet additional composites containing these so-called
7 nanofillers and/or their methods of preparation are described in U.S. Pat.
8 Nos. 4,739,007; 4,618,528; 4,528,235; 4,874,728; 4,889,885; 4,810,734; 4,889,885;
9 4,810,734; and 5,385,776; German Patent 3808623; Japanese Patent J02208358;
10 European Patent applications 0,398,551; 0,358,415; 0,352,042; and 0,398,551;
11 and J. Inclusion Phenomena 5, 473 (1987); Clay Minerals, 23, (1988), 27; Polym.
12 Preprints, 32 (April 1991), 65-66; Polym. Prints, 28, (August 1987), 447-448;
13 and Japan Kokai 76,109,998.

14 Nano clay fillers are also available based on tiny platelets of a special
15 type of surface modified clay called montmorillonite. These surface
16 treatments have been aimed for use with nylon-6 and polypropylene. The
17 two manufacturers in the United States, Nanocor and Southern Clay
18 Products, both point to increases in flexural modulus, heat distortion
19 temperature and barrier properties.

20 Furthermore, attention is hereby directed to U.S. Patent Nos. 5,993,415
21 and 5,998,551 which, although not relating to nano clay fillers, describe the
22 use of crosslinking promoters to improve properties of a thermoplastic
23 material, and, as to be discussed below, are relevant to the present invention.
24 Accordingly, the teachings of these patents are incorporated by reference.

25 In sum, therefore, even with the numerous described composites and
26 methods, it still remains desirable to have an improved composite and
27 method for forming polymer composites derived from a multilayered
28 additive (nano clays) to thereby create composites having improved
29 properties over the polymer on its own.

1 Accordingly, it is an object of this invention to explore the suitability of
2 combining the nano clays with an additional chemical component to establish
3 whether or not the observed mechanical properties of a thermoplastic host
4 resin are improved beyond the use of only a nano clay filler.

5 More specifically, it is an object of this invention to combine nano clays
6 with a suitable crosslinking promotor, and to establish a synergistic effect of
7 such promotors with the nano clay on the mechanical properties of a host
8 thermoplastic matrix.

9 In addition, it is an object of this invention to apply the nano clays and
10 additional chemical component described above (promotor) to develop an
11 improved method to prepare materials suitable for use in medical product
12 applications, such as balloon catheters and catheter shaft production.

13 By way of summary, the present invention comprises a composite
14 comprising a polymer matrix having, dispersed therein, a nano clay in
15 combination with a crosslinking promotor. By use of the term "nano clay" it
16 is noted that such clays are inorganic minerals which have a high aspect ratio
17 with at least one dimension of the particles therein in the nanometer range.
18 By use of the term, "crosslinking promotor" it relates to any chemical
19 compound that will promote crosslinking between those polymer chains that
20 comprise the polymer matrix. Accordingly, it can be appreciate that
21 "crosslinking promotors" include those functionalized chemical compounds
22 that provide the requisite activity, upon activation (irradiation or heat) to
23 chemical react and bond with the polymer chains to form covalent crosslinks
24 between the surrounding polymer chains.

25 Preferably, the crosslinking promotor is trallylisocyanurate or
26 trallylcyanurate, although those skilled in the art will recognize that other
27 types of crosslinking promotors would be suitable and would fall within the
28 broad aspects of this invention. In addition, preferably, the promotor is

1 present in the polymer matrix at a level of about 0.5% to 10% (wt.), and at any
2 increment therebetween in 0.1% increments.

3 As noted, the nano clays are inorganic minerals with a high aspect ratio
4 as one dimension of the particles therein falls in the nanometer range. A
5 variety of references are available to those skilled in the art which discuss and
6 describe nano clays suitable herein. In such regard, the clays having a plate
7 structure and thickness of less than one nanometer are the clays of choice.
8 The length and width of the clays may fall in the micron range. Aspect ratios
9 of the preferred clays are in the 300:1 to 1,500: 1 range. In addition, the
10 surface area of the exfoliated clays is preferably in the range of 700 m²/gram.
11 Nano clays that may be suitable herein include hydrotalcite, montmorillonite,
12 mica fluoride, octasilicate, and mixtures thereof. Nano clay is incorporated
13 herein at a level of 1-10% (wt.) as well as any increment therebetween, in 0.1%
14 increments.

15 Montmorillonite nano clays have a plate like structure with a unit
16 thickness of one nanometer or less. This clay also has an aspect ratio in the
17 1000:1 range. Because montmorillonite clay is hydrophilic, it is not
18 compatible with most polymers and should be chemically modified to make
19 its surface more hydrophobic. The most widely used surface treatments are
20 ammonium cations which can be exchanged for existing cations already on the
21 surface of the clay. The treated clay is then preferably incorporated into the
22 polymer matrix herein, by melt mixing by extrusion, more preferably, twin-
23 screw extrusion. In addition, at such time, and as noted above, the
24 crosslinking promotor can also be readily combined with the clay during the
25 melt mixing process. Those skilled in the art will therefore recognize that, in
26 general, any type of melt mixing process can be applied to prepare the
27 composites of the present invention, including extrusion, direct injection
28 molding, the use of a two-roll mill, etc.

1 With regards to the development of crosslinking herein, as noted, a
2 crosslinking promotor is employed, and preferably, the formulations herein
3 are exposed to irradiation. Preferably, the irradiation dosage is between
4 about 1-20 MR, as well as any numerical value and/or increment therein.

5 In addition, the polymer matrix herein may be selected from any
6 thermoplastic or thermoset type polymer resin host. A representative
7 thermoplastic resin herein is a nylon resin, a nylon block copolymer, nylon
8 block copolymers containing a polyamide block and an elastomeric block,
9 engineering thermoplastic resins (e.g., polycarbonate, polyesters,
10 polysulphones, polyketones, polyetherimides) as well as commodity type
11 materials (polyethylene, polypropylene, polystyrene, poly(vinylchloride))
12 including thermoplastic elastomers. Representative thermoset materials
13 include polyurethanes, epoxy polymers, etc.

14 In method form, the present invention relates to the steps of supplying
15 a polymer matrix, combining said matrix with a nano clay along with a
16 crosslinking promotor. This combination is then preferably exposed to
17 irradiation to develop crosslinking. By the practice of such method, and as
18 can be observed in the various working examples below, a synergistic
19 influence of the promotor has been observed on the ability of the nano clay to
20 improve the mechanical properties of a given polymer matrix. More
21 specifically, in accordance with the invention herein, it has been found that
22 should one combine a given polymer matrix with the nano clay, one will
23 generally observe an increase in mechanical property performance, such as an
24 increase in the flexural modulus. However, it has been found herein that
25 upon incorporation of a crosslinking promotor, the effect of the nano clay is
26 enhanced, in the sense that a synergy is observed as between the promotor
27 and the nano clay on mechanical properties.

28 As a consequence of all the above, the formulations of the present
29 invention are particularly suitable for the development of an intravascular

1 catheter having a tubular shaft comprising a nylon block copolymer and a
2 nano clay filler, including a compound which promotes crosslinking therein,
3 and a soft flexible tubular tip distal of and bonded to said shaft, the
4 improvement comprising irradiation crosslinking said nylon block copolymer
5 of said tubular shaft. The crosslinking is observed to increase the rigidity of
6 the shaft relative to the soft distal tip.

7 In addition, the present invention also relates to a balloon type catheter
8 having a tubular shaft comprising a nylon block copolymer and a nano clay
9 filler, including a compound which promotes crosslinking therein, the
10 improvement comprising irradiation crosslinking said nylon block copolymer
11 of the balloon section.

12 Working Examples

13 First Experiment

14 The first experiment consisted of mixing the Nanocor 130 TGC clay
15 and the southern Clay Closite 30B with Nylon 6 and with Nylon 6 and 3%
16 TAIC. The Nylon 6 used with Allied's Capron B135 WP.

17 The flex modulus did increase with the use of both clays as was
18 anticipated. The increase with the use of a crosslinking promotor was even
19 greater, demonstrating a unique synergy as between the promotor and the
20 nanoclay on mechanical properties. See Table I.

21 Second Experiment

22 The second experiment repeated the first experiment except that the
23 Nylon 6 was replaced by PEBAX® 72 durometer polyamide ether block
24 copolymer. In this case just adding the nano clay did not significantly
25 increase the flex modulus. The surprise was the increase in flex modulus
26 when crosslinking promoters, such as TAIC, was added to the PEBAX® and
27 nano clay. The closite 30B shows the most improvement. A second unique
28 effect was the increase in flex modulus when the combination was
29 crosslinked. In fact the combination of PEBAX®, Closite (nano clay) and

1 TAIC followed by crosslinking more than doubles the flex modulus. See
2 Table II.

3 **Third Experiment**

4 The third experiment was similar to the first experiment noted above
5 except the nylon-6 was replaced by nylon 12, AESNO® from Atochem. The
6 improvements in flex modulus were much like the improvements with the
7 PEBA® in "Experiment Two", noted above. See Table III.

8 **Fourth Experiment**

9 The fourth experiment was similar to the third experiment noted
10 above, except that nylon-12 was replaced by nylon-11, BMNO® from
11 Atcohem. The improvements in flex modulus were much like the
12 improvements with the PEBA® in "Experiment Two". See Table IV.

13 **Fifth Experiment**

14 The fifth experiment was similar to the above, except that both low
15 density and high density polyethylene were employed as the polymer matrix.
16 An improvement in flex modulus was again observed due to the combination
17 of nano clay and promotor (3% wt. TAIC). See Table V.

18

19

NYLON 6				CAPRON B135W			
TABLE I							
NYLON 6	NONE	NANO-130TC	NONE	0	OMR	9,500	150
NYLON 6	NANO-130TC	NANO-130TC	#2	5	OMR	9,500	150
NYLON 6 3% TAIC	NANO-130TC	NANO-130TC	29C	5	OMR	6,200	75
NYLON 6 3% TAIC	NANO-130TC	NANO-130TC	29C	5	5MR	7,200	15
NYLON 6 3% TAIC	NANO-130TC	NANO-130TC	29C	5	10MR	9,500	15
NYLON 6	CLOSITE 30B	CLOSITE 30B	29I	5	OMR	9,400	140
NYLON 6 3% TAIC	CLOSITE 30B	CLOSITE 30B	29B	5	OMR	13,250	190
NYLON 6 3% TAIC	CLOSITE 30B	CLOSITE 30B	29B	5	5MR	10,300	25
NYLON 6 3% TAIC	CLOSITE 30B	CLOSITE 30B	29B	5	10MR	10,100	25
NYLON 6 3% TAIC	NONE	NONE	#1	0	5MR	9,500	50

350,000
450,000
410,000
530,000
550,000
510,000
430,000
550,000
590,000
380,000

PEBAX 7233				PEBAX 7233			
TABLE II							
PEBAX	NONE	CLOSITE 30B	NONE	0	OMR	8,000	250
PEBAX	CLOSITE 30B	CLOSITE 30B	29E	5	OMR	7,800	200
PEBAX 3%TAIC	CLOSITE 30B	CLOSITE 30B	28A	5	OMR	8,500	180
PEBAX 3%TAIC	CLOSITE 30B	CLOSITE 30B	28A	5	5MR	8,500	75
PEBAX 3%TAIC	CLOSITE 30B	CLOSITE 30B	28A	5	10MR	6,500	50
PEBAX 3%TAIC	NANO-130TC	NANO-130TC	28D	5	OMR	9,200	300
PEBAX 3%TAIC	NANO-130TC	NANO-130TC	28D	5	5MR	8,200	150
PEBAX 3%TAIC	NANO-130TC	NANO-130TC	28D	5	10MR	7,800	125
PEBAX 3%TAIC	NONE	NONE	28E	0	5MR	7,900	150

105,000
135,000
160,000
280,000
275,000
135,000
200,000
210,000
150,000

88

TABLE V
NANO CLAY IN HDPE & LDPE

POLYMER	FILLER	FORMULATION	FILLER %	IRA DOSE	BK-STRESS	%STRAIN	FLX-MOD	FLX-MOD % INC
HDPE	NONE	3384	0	0MR	2,317	47	73,802	0
HDPE	CLOSITE 30B	30A	6	0MR	2,231	48	81,580	10.8
HDPE	CLOSITE 30B	30A	6	5MR	1,734	32	94,853	28.9
HDPE	CLOSITE 30B	30A	6	10MR	2,474	29	105,089	42.8
HDPE	CLOSITE 30B	30A	6	15MR	2,886	28	111,028	50.8
HDPE	CLOSITE 30B	30A	6	20MR	3,180	28	113,733	54.5
LDPE	NONE	6005	0	0MR	1,016	49	21,295	0
LDPE	CLOSITE 30B	30B	6	0MR	1,000	46	25,858	21.4
LDPE	CLOSITE 30B	30B	6	5MR	1,434	33	29,339	37.8
LDPE	CLOSITE 30B	30B	6	10MR	1,890	35	31,987	50.2
LDPE	CLOSITE 30B	30B	6	15MR	2,085	38	31,688	48.8
LDPE	CLOSITE 30B	30B	6	20MR	2,034	30	32,884	54.3

1 What is claimed is:

2 1. A polymer composite comprising a polymer matrix having,
3 dispersed therein, a nano clay in combination with a crosslinking promotor.

4 2. The composite of claim 1, wherein said crosslinking promotor is
5 a chemical compound which promotes crosslinking between polymer chains
6 upon exposure to irradiation.

7 3. The composite of claim 1 wherein said crosslinking promotor is
8 triallylisocyanurate or triallylcyanurate.

9 4. The composite of claim 1 wherein said crosslinking promotor is
10 present at a level of about 0.5 to 10 % (wt).

11 5. The composite of claim 1 wherein said nano clay is present at a
12 level of about 1 to 10 % (wt).

13 6. The composite of claim 1 wherein said polymer matrix is a
14 thermoplastic polymer or thermoset polymer.

15 7. The composite of claim 1 wherein said polymer matrix is a
16 polyamide polymer or a polyamide copolymer comprising a polyamide block
17 and an elastomeric block.

18 8. The composite of claim 7 wherein said polyamide block is a
19 nylon-6, nylon-6,6, nylon-11, nylon-12, copolymers of nylon-6/nylon-11,
20 copolymers of nylon-6/nylon-12 or mixtures thereof.

21 9. The composite of claim 7 wherein said elastomeric block is
22 selected from a polyether, polyester, hydrocarbon, polysiloxane or mixtures
23 thereof.

24 10. A composite comprising a polymer matrix having, dispersed
25 therein, a nano clay in combination with a crosslinking promotor, wherein
26 said matrix is irradiation crosslinked.

27 11. A method for enhancing the mechanical properties of a polymer
28 composite, comprising:

29 (a) supplying a polymer matrix;

1 (b) combining said matrix with a nano clay and a crosslinking
2 promotor; and

3 (c) irradiating the combination of step (b) and crosslinking.

4 12. The method of claim 11 wherein said polymer matrix comprises
5 polymer chains, said promotor is a chemical compound that absorbs
6 irradiation and becomes chemically reactive to form crosslinks, and wherein
7 said crosslinks comprise covalent bonds between said polymer chains.

8 13. The method of claim 12, wherein said irradiation is 5, 10, 15 or
9 20 megarads.

10 14. In an intravascular catheter having a tubular shaft comprising a
11 nylon block copolymer and a soft flexible tubular tip distal of and bonded to
12 said shaft, the improvement comprising adding a nano clay filler and a
13 compound which promotes crosslinking therein to said nylon block
14 copolymer forming said shaft, and irradiation crosslinking said nylon block
15 copolymer of said tubular shaft.

16 15. In a balloon type catheter having a tubular shaft comprising a
17 nylon block copolymer and an integrally formed balloon section, the
18 improvement comprising adding a nano clay filler and a compound which
19 promotes crosslinking therein to said nylon block copolymer forming said
20 balloon, and irradiation crosslinking said nylon block copolymer of the
21 balloon section.

22

INTERNATIONAL SEARCH REPORT

International application No.

PCT/US00/31174

A. CLASSIFICATION OF SUBJECT MATTER

IPC(7) : C08J 3/28, 9/00, 9/06; C08K 3/34, 3/20, 3/22; C08L 23/06.

US CL : 522/83, 117, 137; 523/216, 300, 521; 524/445, 447, 449, 451; 604/96, 508, 523.

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 552/83, 117, 137; 523/216, 300, 521; 524/445, 447, 449, 451; 604/96, 508, 523.

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
Please See Continuation Sheet

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 4,444,816 A (RICHARDS et al) 24 April 1984, Abstract, column 1, line 43, to column 2, line 66, column 3, lines 39-45, and Table 3.	1-15
Y	US 4,385,136 A (ANCKER et al) 24 May 1983, column 6, line 26, to column 7, line 49, column 8, lines 5-27, column 10, lines 26-37, Table II, column 11, lines 30-40, column 16, line 30, to column 17, line 10, Examples 1 and 3-6.	1-15
X	US 5,853,886 A (PINNAVAIA et al) 29 December 1998, Abstract, column 3, line 49, to column 4, line 6, column 6, line 13, to column 8, line 18, column 9, lines 4-10, column 9, lines 60-66, column 10, lines 16-26 and lines 57-65, column 13, line 64, to column 14, line 4, column 14, lines 37-47, Examples E4, E13 and E14.	1,5,6,7,10
Y		4,11-13
X	WO 93/11190 (ALLIED-SIGNAL, INC.) 10 June 1993, Abstract, page 7, lines 8-15, column 8, lines 14-20, column 10, lines 26-30, column 21, lines 23-29, pages 23-25, page 28, lines 8-16, page 30, lines 23-29, page 33, line 28, to page 34, line 19, and Example 1.	1,5-8,10
Y		4,11-15
A	US 4,303,595 A (ALLEN) 01 December 1981, Abstract, column 2, lines 56-61, column 3, line 61, to column 4, line 22.	14-15
Y,P	US 5,993,415 A (O,NEIL et al) 30 November 1999, Abstract, column 3, lines 37-58, and column 4, lines 37-50.	1-15



Further documents are listed in the continuation of Box C.



See patent family annex.

* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

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later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

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documents of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&"

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Date of the actual completion of the international search

Date of mailing of the international search report

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INTERNATIONAL SEARCH REPORT

International application No.

PCT/US00/31174

C (Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y,P	US 5,994,445 A (KASHEL et al) 30 November 1999, Abstract, column 3, lines 14-34, column 3, line 53, to column 4, line 27.	1-15
Y,P	US 6,034,163 A (BARBEE et al) 07 March 2000, column 2, lines 22-44, column 3, lines 12-35, column 6, lines 35-61, column 7, lines 52-55.	1-15
Y,P	US 6,136,908 A (LIAO et al) 24 October 2000, Abstract and column 2.	1-15

INTERNATIONAL SEARCH REPORT

International application No.

PCT/US00/31174

Continuation of B. **FIELDS SEARCHED** Item3: USPAT, DERWENT, EPO, JPO: nano clay, nano fillers, nano composites, montmorillonite, hydrotalcite, mica fluoride, ostasilicate, clay, talc, mica, silicate, intercalate, nylon, polyamide, polyethylene, block copolymer